Radionuclide Capture Using

Membrane Technology

Presented at the FETC
Industry Partnerships to Deploy
Environmental Technology Conference

October 1999

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Introduction

3M has developed, with DOE/FETC support, a new technology for selective detection and removal of dissolved radioactive materials from liquids. This technology utilizes particle-loaded membranes fashioned into disks and cartridges, and is intended to provide a cost-effective alternative to present methods of sampling/analysis, and large scale decontamination. The 3M technology provides a novel particle-package with multiple benefits—the use of smaller particles allows for more efficient removal of contaminants at higher flow rates; the membrane/resin matrix prevents channeling of liquids and loss of efficiency; the spiral-wound cartridge offers simplified installation, convenient replacement, and clean, easy disposal of the spent cartridge; the rapid liquid sampler (RLS) allows simple field use of solid phase extraction materials and techniques. Standard, commercially available components are used to incorporate the disks and cartridges into portable, compact systems that can operate at flow rates up to 400 mL/min for disks and from 1 to 50 gallons per minute for cartridges.

Current technologies in use for chemical separations generally are either bulk extraction processes, or column technology systems. These suffer from limitations, especially when applied to large volumes of liquid samples with low levels of target radionuclides, typical in environmental applications. The advantages, flexibility, and applications of membrane technologies developed by 3M, with support from DOE, are described in this paper. The first portion of the paper will discuss 3M membrane technology in general. Later sections will focus on analytical/field sampling applications (3M Rapid Liquid Sampler–RLS), and on remediation cartridge applications. A final section will discuss the research efforts and achievements that are leading to commercialization of membrane technology products.

Particle-Loaded Membrane Technology

In 1989, 3M began introducing particle-loaded membrane products (Empore™) as an alternative form of solid-phase extraction. When used in a filtration-like process, these products captured dissolved species from water for subsequent analysis. Initially, target analytes were primarily organic species. Later, inorganic species and radionuclides were added. In recent years, development of a second membrane technology (WWL™) has produced membranes optimized for cartridge remediation applications.

The unique characteristic of these membranes is their ability to be fabricated with high weight percentages of small, chemically-active, sorbent particles, and still maintain fast flow properties. Particle sizes range from 5 to 80 microns and mass loadings range from 60% to 90%. While densely packed, the membrane structure keeps the particles fixed in place and separated from one another. This promotes easy liquid flow through the membrane with low back pressures. The high surface area of the small particles is readily available, and thus enables fast kinetic interactions between the particle chemistry and dissolved components of the liquid being treated. The net result is a highly efficient separation process. If particles of this size were used in columns, unacceptable back pressures or slow flow rates would result. Channeling and subsequent particle bypass, a common problem with columns, is absent with membranes.

A wide variety of particle types have been successfully incorporated into 3M membranes. The membrane technology allows the use of a number of known high performance chemical-adsorbing particles that previously could not be put into a useful engineered form because of their small particle size. The choice of particle that is selected is driven by several factors. Chemical performance is judged by the capacity of the particle (the degree to which the target compound is adsorbed), and by its selectivity (the degree to which other compounds are adsorbed). Economic considerations for particle selection include the basic cost of the raw material, processing costs, and supply reliability.

Chemistries that are carried by the membrane-bound particles can be quite diverse. In some applications the interacting (adsorbing) molecules are bonded or coated to a particle that is then incorporated into the membrane. In other applications, the particle itself is produced from the sorbent compound. Some general areas that describe feasible particle chemistries are ion exchange resins, coated solid supports, polymeric materials, crystalline molecules, and macrocyclic chelators.

To be effective, membranes must be configured in products that promote easy, user-friendly use. Flat disks cut from the membrane have been used in laboratory analysis applications. These disks have been incorporated into rugged plastic holders and named Rapid Liquid Samplers (RLS) for use in field sampling and analysis applications. Large sheets of membrane are fabricated into cartridges that are used for large volume separations. All of these processes are in pilot plant production level or higher at 3M.

Rapid Liquid Samplers

Rapid Liquid Sampler (RLS) units for cesium (Cs), strontium (Sr), radium (Ra), and technetium (Tc) have been produced and tested successfully. Sampling and analysis procedures have been written for these RLS. Improvements in the physical construction of the RLS have been accomplished, and the commercialization process for their product introduction is well under way. Several methods of field analysis using Empore™ disks housed in RLS have been tested. These results are described further in the following sections. Supporting data and reports are available.

Development of RLS began by testing membranes that were being used in laboratory Empore™ Rad Disk products. Several significant changes in operating conditions for RLS use (as compared with laboratory vacuum filtration methods) were necessary. The two most important changes were operation at fast flow rates and at neutral pH.

In summary, except for Sr, all Rad Disks showed good performance operating under RLS conditions. The Sr Rad Disk did not have sufficient capacity in the presence of some matrix interference ions at a neutral sample pH. An alternate membrane containing the second strontium particle is being evaluated for use in new Sr RLS units.

Table I shows concentrations of seven matrix ions that can be presented to the different RLS membranes and still attain 95% or better retention of the analyte. Matrix interferences are critical in the use of RLS, since poor retention of the analyte will result in data that is biased low. This false negative interference will lead to underestimating, or even not detecting, radioactive contamination. Target matrix concentration levels were chosen based on typical ‘high-level’ surface and groundwater concentrations. The Cs and second Sr membranes exceed all target levels and the Ra membrane exceeds five and is close to two of the target levels. Tc evaluation (done earlier in our contract) shows quantitative recovery at high levels of potential interferents.

Table I
RLS Matrix Interferences
(Quantitative recovery achieved at given ppm level)

Matrix Ion	Target Level	Cs RLS	Ra RLS	2^{nd} Sr RLS	Tc RLS
Na^{+}	100	1000	400	1000	1000
K^{+}	7	1000	3	1000	1000
Mg^{2+}	50	1000	10,000	100	1000
Ca^{2+}	100	1000	10,000	100	1000
Sr^{2+}	1	3	2	20	
Ba^{2+}	0.3	50	0.1	20	
Pb^{2+}	0.1	10	0.3	40	
NO_3^{-}					3000
Cl^{-}					100,000

Radiometric inferences need to be minimized with RLS use so that reliable data is obtained. However, especially in water monitoring applications, radiometric interferences are not as critical as matrix interferences since they will produce false positive results. If these results are above an action threshold, then further testing can be instigated. Tested radiometric interferences are summarized in Table II.

Table II
RLS Radiometric Interferences

Radionuclide	Cs RLS	Ra RLS	2^{nd} Sr RLS
Ba-133	-	-	-
Co-60	-	++	-
Cs-137		-	--
Ra-226	--		-
Sr-85	-	--	
Y-90	-	+	-
Tc-99	++	++	++
U-238	+	+	-
Pu-239	++	+	-

<u>Interference Retention Key</u>

++	<10%
+	10-50%
-	50-90%
--	>90%

Flow rate of a sample through the RLS membrane needs to be fast enough to keep the overall sampling time reasonable for a person taking a large volume sample (e.g., four liters). The specified minimum flow is 200 mL/min. Figure 1 shows the relationship between flow rate and analyte percent recovery. Earlier work has shown no loss of Tc at flow rates up to 400 mL/min. All RLS can be used successfully at any flow rate up to 300 mL/min.

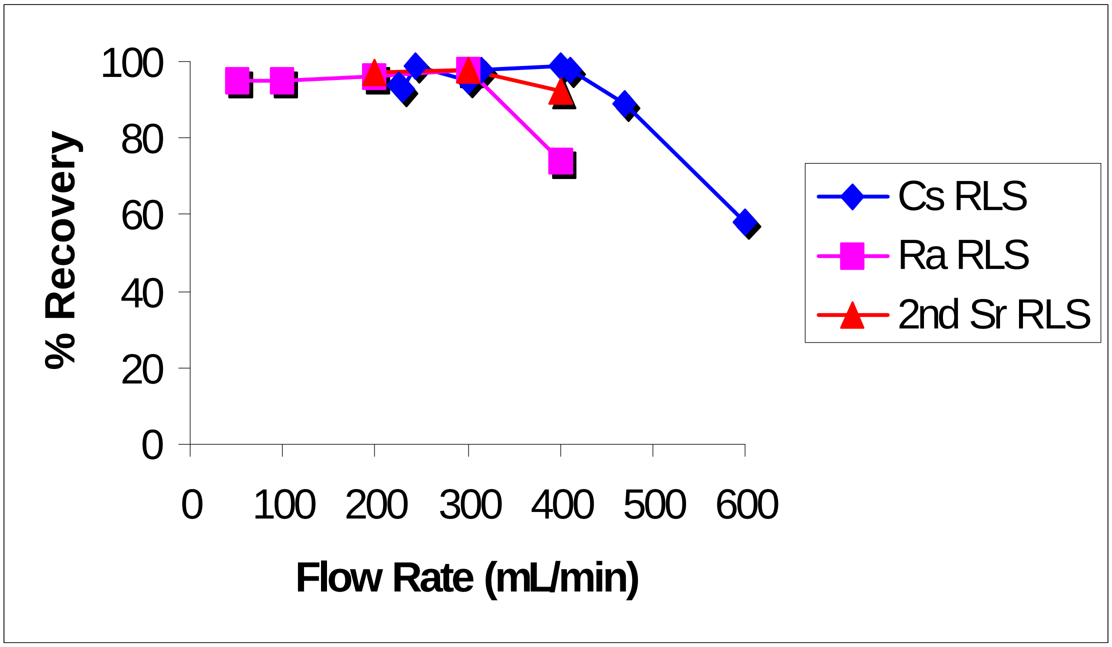

Figure 1. RLS flow rate testing.

All RLS types have been subjected to three ruggedness tests. The first of these is shelf-life temperature testing. Results show that these units may be subjected to temperatures of 0°C to 60°C (32°F to 140ºF) for thirty days and still perform as specified. Another temperature test processed sample waters at two temperatures—<5°C and >40°C. Results show greater than 90% analyte recovery for all RLS types, except the Sr RLS processing cold water. Holding time testing shows all RLS are stable for at least 30 days after processing a sample.

The Rapid Liquid Sampler consists of an Empore™ disk, a scrim material that back the Empore™ disk, and the plastic holder that encloses the scrim and the disk. The holder and scrim must be constructed to withstand the pressure generated during the entire time the RLS is processing a sample. The current goal for this pressure is 50 psi. A second requirement is that the disk must be easily retrievable from the holder after sample loading. A third major requirement is that the cost of production be low enough to promote commercial development and customer acceptance of the final product.

During 1997, an article that satisfied the 50 psi requirement was attained. In order to easily open the RLS, a portable manual press with a cutting die was designed and produced. Although this device performed well, it did not satisfy the third requirement for commercial development. Consequently, in 1998 and 1999, work proceeded on improving the design of the RLS to satisfy all three requirements.

Simpler alternative devices to open the RLS were researched, designed, and fabricated. Alternative RLS overmold materials were tested. Often it was found that materials that were easier to open did not satisfy the pressure requirement. An improved design of the injection molding tool was completed, but not built, due to high cost concerns, and the timely successful identification of an overmold material that can be cut apart manually. Slight reworking of the existing tool incorporated some of these improvements at low cost.

<u>Burst Testing</u>

The goal of burst testing was to determine the best material for overmolding the RLS. The best material will seal the RLS to withstand operating pressures, and yet be easy to open for analysis. Several candidates were evaluated initially by pressurizing the RLS with air and looking for leaks while holding the RLS under water. Three candidates were selected from this initial group for dynamic flow testing. In dynamic flow testing, water is pumped through the RLS in a system that generates 60 psi of back pressure. One candidate was chosen for final testing using cold and hot water (4°C/40°C) in the dynamic flow test, simulating potential field condition extremes.

<u>Scrim Testing</u>

The goal of scrim testing was to determine the best scrim material for use within the RLS, and to determine if the scrim needs to cover the inlet and outlet faces of the chemically selective membrane. The best scrim will support the membrane within the RLS, allow the membrane to capture a maximum of analyte, allow a high flow rate through the RLS, and not retain chemical species itself. Three scrim materials were selected from several candidates for extensive testing. These were tested by placing one scrim on both the inlet and outlet side of the membrane, or one scrim on the outlet side only. Studies showed that all configurations successfully provided good analyte retention, good flow rates through the RLS, and good compatibility with liquid scintillation radiometric counting.

<u>RLS Use</u>

Rapid Liquid Samplers have been designed to be usable with any pumping system. Operational information including pressure and flow rate specifications are documented in the methods for use for each RLS.

An attended pump system that uses a portable, battery-powered, peristaltic pump is one way the RLS can be used. This system can be augmented with prefiltration filters or capsules, pressure gauges, and/or sample measurement devices. If volumes less than one liter are to be sampled, a manual pump is often satisfactory. For smaller volumes, large syringes with luer fittings connect very simply to the RLS.

RLS have also been used in conjunction with 3M cartridge remediation systems to monitor the performance of the cartridges. Sampling ports with valves and luer lock adapters built into the cartridge system allow easy sampling and monitoring of the remediation process with appropriate Rapid Liquid Samplers.

Another system that is using RLS is the time or flow composite sampler. Isco, Inc. has modified its market-leading Model 3710 Sampler to use RLS. (The new model is 3710 SPX Sampler.) Savannah River Technology Center (SRTC) is using these samplers in its monitoring at the Savannah River Site. In addition, DOE is funding current deployment of these samplers at other DOE sites through an Accelerated Site Technology Deployment (ASTD) project.

RLS Applications

Test methods for the use of newly developed RLS for field sampling and subsequent conventional laboratory radiocounting have been written. Early development work exploring alternative counting equipment and procedures has focused on three areas: direct field radiocounting of RLS disks, colorimetric reactions on disks, and scintillation disk development.

Direct Field Radiocounting

In the area of direct field radiocounting, studies using commercially available instrumentation from Oxford Instruments, Inc. have been performed in our laboratory. Excellent correlation between data from the counting of disks by a Tennelec Solo instrument (a field-transportable low background gas proportional counter) and a conventional laboratory low background gas proportional counter have been achieved. Correlation of Cs RLS counts by the field portable Nanospec gamma spectrometer to results from a laboratory high-purity germanium gamma spectrometer have shown a strong linear relationship.

In addition to testing RLS membranes with commercially available instrumentation, work is underway to assemble less expensive instrumentation that can take advantage of disk selectivity. The DOE Environmental Measurements Laboratory (EML) has been working on developing an inexpensive portable detector used to quantitate activity on a Cs membrane. The detection system consists of a sodium iodide crystal combined with a photomultiplier tube and a preamplifier. The temperature dependence of this system was investigated (significant above 70°F, with up to 10% decrease in response). EML is working on means of correcting for this change, with the most promising proposal consisting of using an internal check source, which could be used to lock on the signal and compensate for the drift due to the increasing temperature. The extent of shielding is also being evaluated, in order to balance the needs of portability, strength of the check source, and analytical sensitivity required. A microcomputer is in place and interfaced with the display. Work is in progress to interface that same microcomputer with the detector.

Colorimetric Reactions

In the area of investigation into colorimetric development of loaded Empore™ disks, work has continued with the lead disk. Here, an assay that has good sensitivity and is a fast analysis has been developed. The iron interference for the lead disk reported in the Phase I can now be counteracted through careful pH adjustment. The colorimetric test can detect micrograms of dissolved lead in the presence of milligrams of dissolved iron. Using a colorimeter for color measurements, a calibration curve for quantifying lead in the range of 20 to 50 µg/L in the presence of approximately 2000 µg/L (2 mg/L) dissolved iron has been produced.

Scintillation Disks

Scintillation disks are Empore™ disks that contain both selective absorbing particles and particles of a solid scintillator. Careful membrane construction to optimize the close proximity of these particles results in good efficiences. A linear correlation between the activity of technetium-99 loaded onto a disk and the response of a portable photomultiplier detector has been demonstrated. The efficiency shown by this system is about 20%. Lower activities (below ~500 pCi) deviated from a good linear response mainly due to high background levels. Identification of better instrumentation should improve the sensitivity. Note however, that the activity on the curve would be a lower activity per liter when a multi-liter sample is processed (e.g., 500 pCi on a disk that had processed 5L of sample would give a result of 100 pCi/L in the original water sample.)

Cartridge Remediation

The 3M Selective Separations Remediation Cartridge may be considered a combination of the sciences of particle chemistry and membrane technology coupled with innovative article and system design. These important features of the cartridge technology are discussed below.

Particle Chemistry

The Selective Separation Cartridge technology relies on the use of chemically active particles that have the ability to adsorb specific dissolved materials from liquid media. The criteria for selecting particles for use in cartridges include:

- Affinity for the target analyte, i.e., it must be able to capture and hold the radionuclide of concern;
- Capacity for retention of the target analyte, i.e., it must have sufficient capacity on a per weight basis to make it operationally useful;
- Physical suitability, the particle must be able to be incorporated into a membrane with acceptable functional characteristics with regards to flow rate, pressure drop, and kinetic performance;
- Economically feasible – the particle must be available in a cost range that makes the final cost of the complete system competitive with existing technologies.

3M has examined particles for a variety of radionuclides, including cesium, strontium, technetium, uranium, plutonium, radium, and cobalt. Particles useful for the capture of each of these elements have been put into membranes and tested in the lab for efficiency and retention capacity. A typical performance curve will evaluate affinity for the target analyte in a matrix containing competing ions, and will plot percentage of analyte breakthrough as a function of gallons of water processed. A performance curve for strontium absorption in the presence of sodium, potassium, and calcium, is shown in Figure 2.

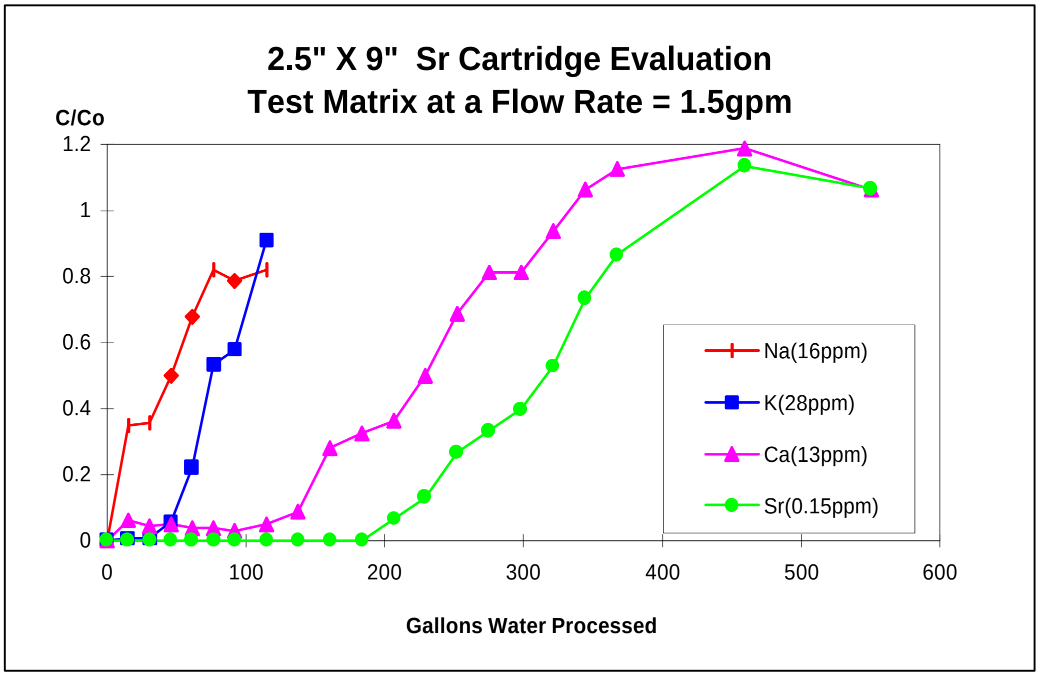

Figure 2. Strontium cartridge performance

Figure 2 shows that sodium and potassium break through quickly, and that calcium breaks through before the strontium. This is the desired scenario, because it indicates the particle's affinity for the strontium ion over the other ions; the particle will cling most tenaciously to strontium when challenged with a mixed solution. In this case, a flow rate of 1.5 gallons per minute was used in testing a 2.5" diameter by 9" length cartridge. A larger (6" x 22") cartridge was used in a test at a higher flow rate to obtain the results shown in Figure 3. Figure 3 also illustrates that the particle has greater affinity for strontium than calcium.

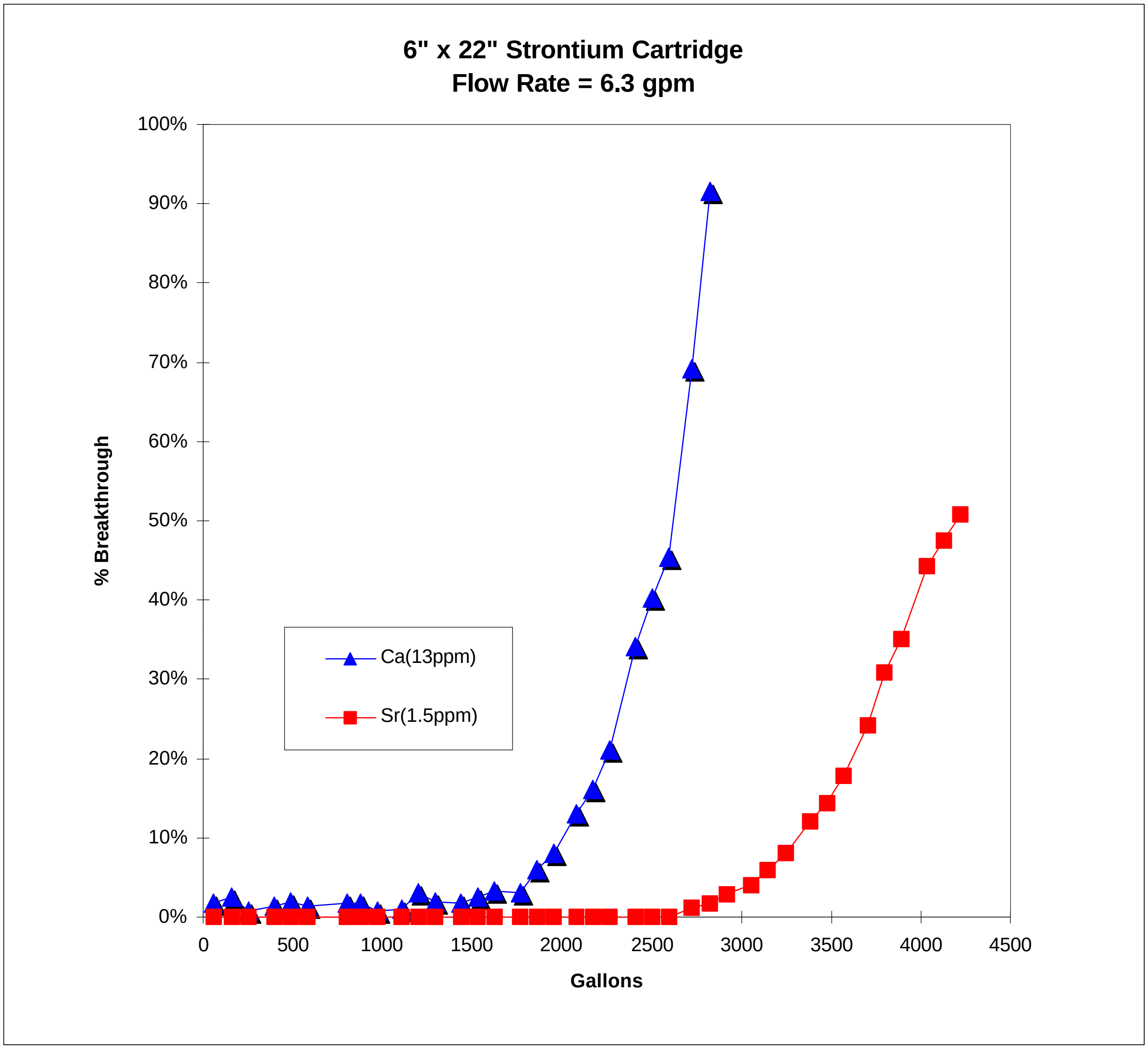

Figure 3. Large strontium cartridge performance

<u>Membranes</u>

The 3M WWL™ membrane is prepared using a wet-laid process similar to paper-making; particles and fibers are combined with a suitable binder to form a flat, continuous sheet of material. At the time of membrane manufacture, formulation and processing details are carefully monitored and/or adjusted to produce desirable characteristics in the finished membrane. Because of the many different radionuclides being targeted in the DOE cleanup effort, a number of different particles and particle types are ultimately incorporated into membranes, and each particle calls for specific formulation and processing conditions. Pilot-scale batch processing currently produces from several hundred to several thousand feet of usable membrane at a time. The finished membranes are rolled up onto large cores and stored as 'jumbos.'

Cartridges

Each 3M Selective Separation cartridge is fabricated from a WWL™ membrane jumbo roll by converting the membrane into a shorter, uniform sheet, combining it with a scrim material, and winding it onto a hollow, perforated core. The formed article is referred to as a spiral-wound cartridge. The ends are then capped – one closed, the other open – to form the final article. The direction of flow through the cartridge is from outside to inside. Water surrounds the cartridge in its housing, passes through the layers of membrane, through the perforated core, and then out of the open end. Removal of the target radionuclide takes place as water passes across the active particles in the membrane layers.

Separation cartridges may be prepared in a number of different sizes; a typical cartridge designed to process liquid at 1 to 2 gallons per minute measures 3” in diameter by 10” in length, and contains approximately 450 square inches of membrane with from 150 to 250 grams of available active particle. Larger cartridges have been designed to handle increased flow rates; an experimental cartridge measuring 6” in diameter by 21” in length was used to process water at a flow rate exceeding 15 gallons per minute. Cartridge dimensions are predicated both on system requirements with regards to flow and back pressure and industry standards for filter vessels and housings. For instance, the open ends of the cartridges are fitted with o-rings that adapt to one of two industry-standard receptacles for filters, designated as 222 and 226. It is possible that a cartridge could be made to dimensions other than those typical to the industry, but this would require specialized equipment and fittings, and would result in increased system costs.

Systems

The 3M Selective Separation cartridges are deployed in a system that includes a pump to provide water flow, filter vessels for containment of the active cartridges, appropriate valves and gauges for controlling and monitoring flow and pressure, plumbing to direct influent and effluent streams, and utility carts/tables to carry the system components. Because the requirements for each application vary, there are a variety of systems that may be provided with the cartridges. The simplest system is an arrangement of vessels in series: water enters at one end and passes through a vessel (or series of vessels) containing prefilters for solids removal, and then through active cartridges for radionuclide removal, exiting the opposite end of the system. Vessels may contain single cartridges or a multiple of cartridges. This latter arrangement is referred to as a ‘nested’ system. More advanced systems allow for the redirection of flow through the active vessels so that cartridges can be replaced in a manner that provides maximum remediative efficiency. The combination of large, nested cartridges in a redirectable flow system provides high flow rate in a continuous operation mode.

Site Technology Demonstrations

3M is currently conducting two demonstrations of the cartridge technology at DOE sites in Ashtabula, Ohio and Savannah River Site (SRS), South Carolina. Several commercial ventures are ongoing at Brookhaven National Laboratory (BNL), New York.

Removal of technetium from groundwater at a flow rate of 10 gpm is being demonstrated at the RMI Titanium plant in Ashtabula, Ohio. The system has been used to process approximately 20,000 gallons of water to date. Influent levels of Tc-99 from 1500 to 8000 pCi/L have been reduced to 450 pCi/L or less throughout the demonstration.

The second demonstration is being prepared at the R-disassembly basin at SRS, for removal of Sr-90 and Cs-137. A flow rate of 25 gpm is targeted and the test will be run in two parts, as each of the radionuclides requires a different cartridge. Equipment is on-site and awaiting final site modifications.

At BNL, 3M installed and operated a small, portable filtration system designed for removal of Sr-90 from tank water at the site. Using three cartridges arranged in sequence, Sr-90 levels were reduced from 20 pCi/L to less than half of the administrative release limit of 4 pCi/L. RLS were used to monitor influent and effluent strontium levels. The system remains on site, available for continued use as needed. A second system was delivered, installed, and operated for removal of Pb and Co to below action limits in water from a waste storage basin.

<u>Previous Demonstrations</u> (1995-1997)
Paducah – Removal of technetium from groundwater was accomplished using a single 7-inch cartridge and a flow rate of 1 gallon per minute. Twenty-two thousand (22,000) gallons of water were treated and Tc-99 levels were reduced from 1000 pCi/L to 24 pCi/L.

SRS – A seven-day test demonstrated removal of Cs-137 from 55,000 gallons of water from the R-disassembly basin at Savannah River Site. Water was processed at 6 gpm and cesium activity was reduced from about 83,000 pCi/L to detection limits over the course of the test.

N-Basin, Hanford – Cs-137 was removed from 375 gallons of water at a flow rate of 0.8 gpm. Activity level was reduced from 124,000 pCi/L to 36 pCi/L.

Argonne National Laboratory – Cs-137 and Co-60 were removed from 4500 gallons of water at the CP-5 reactor site. This study is detailed in the Green Book of DOE Innovative Technology Summary Reports.

Additional smaller scale demonstrations were conducted at INEL, Idaho Chemical Processing Plant (Sr-90 and Cs-137), West Valley (Tc-99 and Cs-137 removal), and Idaho Falls, Test Area North (Cs-137 and Sr-90 removal).

Moving to Commercialization

Research and development work and subsequent promising technologies must move on to become commercially viable products. This process is successful only when efforts in areas such as manufacturing, marketing, and technical support are strong. Products for radionuclide capture that use 3M membrane technology are moving rapidly through the commercialization process. Rad disks have been commercially available for several years. Certain RLS units will be commercially introduced early in 2000. Sales of experimental remediation cartridges are testing the market and providing feedback from real end-users.

Combined Research and Development Approach

Although the applications for membrane technology fall into two major product categories (disk products and cartridge products), research and development efforts are being conducted together. Investigation of the chemistries and the availability of particles is a similar effort for both types of products. In addition, the analytical support (both radiochemistry and elemental analysis) required to evaluate particles, membranes, disks, and cartridges is the same. Field demonstration and deployment can also occur together.

Collaborators

Development of this technology and these products has been enhanced by collaboration with many different groups and individuals, both within and outside of 3M. Some examples of recent collaborators include DOE sites and laboratories (Savannah River Site, Argonne National Laboratory, Brookhaven National Laboratory, Environmental Measurements Laboratory, and Los Alamos National Laboratory), other governmental groups (EPA), universities (University of Georgia, University of California-Berkeley), contract laboratories (Paragon Analytics Inc.), and instrument companies (Packard Instrument Company, Isco Inc., and Oxford Instruments Inc.).

Technology Achievements

Products developed with this membrane technology have been honored with three significant awards:

- R&D Magazine's award for One of the 100 Most Technologically Significant New Products of the Year 1996
- 1997 Federal Laboratory Consortium of Technology Transfer Award of Excellence
- American Filtration and Separations Society 1998 Award for New Product of the Year

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